Low-Volume and Ultralow-Volume Sprays of Malathion and Methyl Parathion for Control of Three Lepidopterous Cotton Pests

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CONTENTS

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Low-Volume and Ultralow-Volume Sprays of Malathion and Methyl Parathion for Control of Three Lepidopterous Cotton Pests

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SUMMARY

Methyl parathion in low-volume (LV) spray was more effective than malathion against larvae of tobacco budworm and bollworm in the laboratory. In topical applications, malathion and methyl parathion were more toxic than malaoxon and methyl paraoxon to the tobacco budworm and the bollworm. Topical applications of malathion, malaoxon, methyl parathion, and methyl paraoxon killed greater percentages of larvae of the bollworm than of the tobacco budworm.

In one field experiment, ultralow-volume (ULV) sprays of malathion and methyl parathion killed 85 percent of bollworms and tobacco budworms. Malathion in ULV or LV sprays in two other experiments reduced larval populations only 0 and 23 percent. Methyl parathion adequately controlled these insects. It is doubtful that malathion could give effective control, since its effectiveness varied in the three field experiments. Methyl parathion was effective against the pink bollworm in the laboratory and field; malathion was effective in laboratory experiments but not in the field.

Gas chromatography was used to determine malathion and methyl parathion qualitatively (comparing R_t of sample and standard from the solvent peak) and quantitatively (peak height of sample compared with peak height of a standard over the range of linearity). Recoveries of malathion and methyl parathion were 75 to 95 percent.

More methyl parathion was found on the cotton leaf when the compounds were applied in ULV than in LV sprays.

INTRODUCTION

Malathion and methyl parathion were first evaluated in the early 1950's as conventional low-volume (LV)[1] sprays for cotton-insect control. The use of these compounds has increased since 1964 when technical malathion, applied in an ultralow-volume (ULV)[1] spray, was shown to be effective against the boll weevil, *Anthonomus grandis* Boheman (*4*, *15*).[2]

ULV sprays of malathion were not effective against the bollworm, *Heliothis zea* (Boddie), and tobacco budworm, *H. virescens* (F.), in Texas, Arizona, and Mississippi (*1*, *3*, *16*), but Hopkins and Taft (*8*) stated that malathion was effective against these insects in South Carolina. Methyl parathion has controlled boll weevil populations in the field (*11*) and the boll weevil, the bollworm, and the tobacco budworm in the laboratory (*2*, *3*, *9*, *13*). McGarr and Wolfenbarger (*12*) demonstrated that ULV sprays of methyl parathion are effective against the bollworm and the tobacco budworm in the field. Information has not been published on the effectiveness of these insecticides in ULV sprays against the pink bollworm, *Pectinophora gossypiella* (Saunders).

Wheeler et al. (*17*) showed that malathion applied in ULV sprays persisted longer than in LV sprays. Nemec et al. (*14*) showed that greater deposits of methyl parathion were found and that residues persisted longer when it was applied in ULV than in LV sprays. Further information was needed on the comparative effectiveness against cotton insects of these compounds when applied in LV and ULV sprays.

To get the desired information, malathion and

[1] Abbreviations for methods of application used throughout this report are as follows: LV—low volume, more than 64 ounces of total solution diluted in water applied per acre; ULV—ultralow volume, 64 ounces or less of total solution applied per acre.

[2] Italic numbers in parentheses refer to Literature Cited, p. 13.

methyl parathion in LV and ULV sprays were evaluated in the field at Altamira, in Tamaulipas, Mexico, and at Brownsville, Los Fresnos, Progreso, and Bayview in Texas. Malathion and malaoxon (*O,O*-dimethyl (S-[1,2-*bis*(ethoxycarbonyl)ethyl] phosphorothiolate)), methyl parathion, and methyl paraoxon (*O-O*-dimethyl *p*-nitrophenyl phosphate) were compared for toxicity to larvae of bollworm and tobacco budworm and adults of pink bollworm in the laboratory. We considered it necessary also to determine effectiveness of malaoxon and methyl paraoxon because they are the most important oxidative *in vivo* cholinesterase inhibitors (*7*) of malathion and methyl parathion.

We also obtained information on drift of methyl parathion when applied in ULV and LV sprays and effectiveness of different swath widths of ULV sprays when applied by airplane. Combined with the data on effectiveness against insects, this information could give us a better understanding of how ULV sprays should be applied to cotton plants for insect control.

Gas-liquid chromatographic (GLC) techniques have previously been reported for determining malathion and methyl parathion concentrations (*5, 6, 10*). We used these GLC techniques to detect malathion and methyl parathion concentrations from cotton leaves.

PROCEDURES, DISCUSSION, AND RESULTS

Effectiveness in laboratory tests

Mortalities among 30 to 60 larvae of bollworm and tobacco budworm and 20 to 40 adults of pink bollworm were determined 48 hours after malathion, malaoxon, methyl parathion, or methyl paraoxon were applied in LV and ULV sprays to cotton plants, using techniques described by Wolfenbarger et al. (*20*) and Wolfenbarger and Lowry (*18*). These techniques are not described in this report.

Initial toxicities were expressed as lethal concentrations (LC) in pounds per acre to kill 80 percent of the treated larvae or as percentage mortalities at indicated concentrations. Residual toxicities were determined by exposing larvae or adults to treated plants on certain days after application of the sprays. The LV sprays were applied to plants on a turntable, and ULV sprays with a traveling boom sprayer inside a chamber.

Topical applications of malathion, malaoxon, methyl parathion, and methyl paraoxon in 1 microliter acetone were made to the dorsum on the midthoracic segment of 50 to 60 4- to 5-day-old tobacco budworm larvae for LD_{50}'s (lethal doses sufficient to kill 50 percent of the larvae). Mortalities were determined 48 hours after application and are expressed as milligrams of toxicant per gram of body weight from eye-fitted lines according to Wolfenbarger and Redfern (*21*).

Malathion and malaoxon

For LC_{80}'s against tobacco budworm and bollworm, malathion in an LV spray had to be applied at 3.84 pounds and 1.77 pounds per acre, respectively. The amount of malaoxon required was more than 10.0 pounds per acre for the tobacco budworm and 1.77 pounds for the bollworm. Malathion at 2.5 pounds per acre in ULV and LV sprays gave kills of 64 and 60 percent of tobacco budworms and bollworms, respectively. Malathion applied at 2.0 pounds per acre in LV spray killed 100 percent of pink bollworm adults 48 hours after application. This compared with a kill of 95 percent when applied at 1.81 pounds per acre in a ULV spray. Malathion, when applied in LV spray, killed 70 percent of pink bollworm adults after 2 and 8 days; when applied in ULV spray, the kill after 2 and 8 days was 77 and 31 percent, respectively.

In topical applications, LD_{50}'s of malathion were 0.018 and 0.0043 mg. per gram for larvae of tobacco budworm and bollworm, respectively. LD_{50}'s of malaoxon were 0.090 and 0.043 mg. per gram for larvae of tobacco budworm and bollworm, respectively. Thus, malathion was more toxic than malaoxon when topically applied. Since the oxons are more potent cholinesterase inhibitors than the thio moiety, we suggest that the oxidized compounds do not penetrate the cuticle as well as their thio analogues.

Methyl parathion and methyl paraoxon

Methyl parathion applied in LV sprays at 0.25 and 0.50 pound per acre killed 100 percent of bollworm and tobacco budworm larvae. Methyl paraoxon at 1.0 pound per acre killed 100 percent of tobacco budworm, and at 0.5 pound per acre it killed 85 percent of bollworm larvae. Methyl parathion applied in ULV and LV sprays at 1.13 pounds per acre killed 100 percent of tobacco budworm larvae within the first day; the kill after 2 days for ULV spray was 60 percent and for LV spray 18 percent. Methyl parathion in LV spray at 0.125 pound per acre killed 100 percent of pink bollworm adults. At 0.5 pound per acre, after 1 and 3 days it gave kills of 33 and 23 percent of pink bollworm moths, respectively.

When methyl parathion was applied topically to tobacco budworm and bollworm larvae,

LD_{50}'s were 0.043 and 0.030 mg. per gram, respectively.

Effectiveness against insects in field cages

Malathion (5 pounds per gallon emulsifiable concentrate) and methyl parathion (4 pounds per gallon emulsifiable concentrate) were applied in LV sprays to field-grown cotton plants in cylindrical field cages, as described by Wolfenbarger et al. (*20*). Twenty-five tobacco budworm larvae were used per concentration in 1965 and 1966, and 50 in 1967.

Malathion at 2.5 pounds per acre killed no larvae of tobacco budworm. Methyl parathion at 0.5 and 1.0 pound per acre killed 51 and 80 percent of larvae of tobacco budworm, respectively.

Effectiveness in field tests

Technical malathion (9.7 pounds per gallon) was applied in a ULV spray; formulated malathion (5 pounds per gallon emulsifiable concentrate), and toxaphene plus DDT plus methyl parathion (4 pounds of toxaphene plus 2 pounds of DDT plus 1 pound of methyl parathion per gallon emulsifiable concentrate) were applied in LV sprays. Insecticides were applied with an airplane (Ag-Cat[3] Model 2500) at Progreso, Tex. in 1965. Treatments were replicated three times. Each plot was 400 feet long and 50 feet wide for ULV and 35 feet wide for LV sprays. Six Mini-Spin nozzles (manufactured by Buffalo Turbine Company) were used to apply malathion in ULV sprays. The LV spray was applied from a height of 6 feet at 3 gallons per acre, and the ULV spray was applied at a height of 10 feet at 1 pint and 1 quart per acre.

Efficacy of the sprays was determined by placing 5 larvae of tobacco budworm or bollworm (5 to 7 days old) on 4 or 5 leaves collected from each plot in each of five 9-cm. petri dishes (1 dish/replicate) in the laboratory. Mortalities were recorded as described by Wolfenbarger et al. (*20*) for LV sprays in the laboratory. Larvae were reared on an artificial diet in 1-ounce cups prior to the bioassays in the laboratory. Mortalities were averaged on 0, 1 to 2, 4 to 5, and 6 to 7 days after the first, second, fourth, sixth, eighth, and tenth applications.

Immediately after malathion was applied in ULV and LV sprays, mortalities of 85 to 90 percent were obtained on leaves from the top of the plant and 38 to 40 percent on leaves from the bottom of the plant (table 1). The average differences in mortality on leaves sampled at the bottom and top of the plant were 49 percent when tested immediately, 29 on 1 to 2 days, and 13 on 4 to 5 days; after 6 to 7 days the difference was slightly in favor of the bottom leaves.

In a test at Brownsville in 1966, 14 applications of malathion in an LV spray at 2.5 pounds per acre were made with a high-clearance sprayer to plots of cotton between July 10 and October 13. Plots were 1/11th acre in size with four replicates. They were compared with an untreated check. One hundred cotton squares were examined per plot for larvae and damage. The average percentage of squares injured and numbers of larvae per 100 squares were 56.1 and 18.9 for the malathion-treated plots and 71.4 and 24.8 for the untreated check.

In 1967, malathion and methyl parathion applied in ULV and LV sprays with ground equipment were compared for effectiveness against bollworm, tobacco budworm, and pink bollworm. ULV sprays were applied with a John Blue mist-blower sprayer mounted on a high-clearance tractor which has two air outlets with a fan nozzle in the center of each to disperse the spray. Four rows were sprayed from each outlet. LV sprays were applied at 5 gallons per acre with a 12-row high-clearance sprayer equipped with 3× nozzles spaced 20 inches apart on the boom. Plots were 1/11th acre in size (12 rows wide) and treatments were replicated four times. Eight applications were made at 5- to 7-day intervals. One hundred squares and/or bolls were examined per plot at about weekly intervals to determine control of the tobacco budworm and the bollworm, 100 squares for the boll weevil, and 50 bolls for the pink bollworm.

TABLE 1.—*Toxicity of malathion and toxaphene plus DDT plus methyl parathion to bollworm and tobacco budworm larvae, Progreso, Tex., 1965*

Insecticide (lb./acre)	Type of spray	Location of leaf sample on plant	Percentage mortality on indicated days after application			
			0	1–2	4–5	6–7
Toxaphene 3.0 + DDT 1.5 + methyl parathion 0.5.	LV	Top	85	55	41	37
		Bottom	41	23	25	32
Malathion 2.5	ULV	Top	90	48	32	23
		Bottom	40	18	19	33
Do	LV	Top	85	45	28	27
		Bottom	38	18	16	29

[3] Trade names are used in this publication solely for the purpose of providing specific information. Mention of a trade name does not constitute a guarantee or warranty of the product by the U.S. Department of Agriculture or an endorsement by the Department over other products not mentioned.

Methyl parathion and malathion applied in a ULV spray were significantly more effective against bollworm, tobacco budworm, and pink bollworm than when applied in LV sprays (table 2). Methyl parathion was more effective than malathion as a ULV spray against all three insects. Malathion was ineffective against pink bollworm populations.

Methyl parathion was applied in a ULV spray at 1.0 pound per acre in five swaths each 53 feet (16 rows), 66 feet (20 rows), and 79 feet (24 rows) wide. A Snow airplane equipped with 8 Mini-Spin nozzles was used to apply the toxicant at an altitude of about 6 feet. Seventeen applications were made at intervals of 5 to 7 days. Examination was made of 300 squares and a like number of bolls for injury by the tobacco budworm and bollworm and for punctures by the boll weevil in the middle and downwind portions of the swath widths. A 1-acre untreated area on the upwind side was used as a check and was examined as described above.

Methyl parathion was about equally effective in all swath widths against the tobacco budworm and the bollworm. Seasonal averages for damaged squares (12 records) and bolls injured (14 records) ranged from 2.0 to 3.9 percent for treated plots compared with 17.5 percent for the untreated plot. Squares punctured by the boll weevil ranged from 0.3 to 1.1 percent compared with 4.5 percent for the untreated. Increases in yield from the three swath widths over the untreated ranged from 1,124 pounds for the 16-row to 1,400 pounds for the 20-row swath widths. The yield for the untreated was 1,351 pounds. Yields were based on boll counts, with 100 bolls estimated to equal 1 pound of seed cotton.

Effectiveness of methyl parathion applied in different swath widths was bioassayed by selecting 40 to 50 leaves taken 0, 1, 3, 4, 6, and 8 days after three or more applications from treated plots on the downwind (SE) and upwind (NW) sides from the plot center as well as from the plot center. Larvae of tobacco budworm were placed in five petri dishes (five larvae per dish) with treated leaves brought from the field to the laboratory. Mortalities were recorded after 48 hours. Larvae were placed on leaves from greenhouse-grown plants for check mortalities. There was little difference (7 to 8 percent) in larval kill after 8 days between the downwind and upwind side of the plots and plot center (table 3). This indicates that ULV sprays of methyl parathion applied by airplane were equally toxic regardless of whether the swath width was 16, 20, or 24 rows.

Deposits of methyl parathion in laboratory and field tests

Residues of methyl parathion applied in LV and ULV sprays to cotton plants were determined by GLC techniques by methods of Wolfenbarger et al. (*19*). Qualitative information was determined by retention time (R_t) with standards. The R_t was 2 to 4 minutes under the indicated operating conditions. Concentrations that "pegged" (exceeded 100 percent full scale) or exceeded 50 percent of full scale (electron-capture detector) were diluted to prevent peak height from departing from linearity. Full-scale peak height was employed for the sodium thermionic detector. Injections of 1 to 5 μl. of sample were made into the evaporation chamber, and two or more injections were made for all samples. Averages were obtained from two or more peak heights. Standards were run between

TABLE 2.—*Effectiveness of malathion and methyl parathion against bollworm, tobacco budworm, and pink bollworm, Brownsville, Tex., 1967*

Insecticide (lb./acre)	Type of spray	Bollworm and tobacco budworm		Pink bollworm	Seed cotton per acre
		Damaged squares	Larvae	Bolls infested	
		Percent [1]	*Number*	*Percent*	*Pounds*
Methyl parathion 1.0	ULV	5.3 a	1.0 a	1.4 a	2020 a
Do	LV	7.8 d	1.9 c	4.0 b	1907 a
Malathion 2.5	ULV	5.7 b	1.3 b	6.8 c	1905 a
Do	LV	7.0 c	1.8 c	13.8 e	1744 a
Check		10.6 e	3.2 d	10.8 d	1331 a

[1] Figures followed by the same letter are not significantly different from each other as determined by Duncan's multiple range test at 5-percent level.

TABLE 3.—*Mortality of tobacco budworm larvae after 48 hours in laboratory bioassays of leaves collected from plots in which methyl parathion was applied in ULV sprays in different swath widths at 1.0 pound per acre, Los Indios, Tex., 1966*

Days after application	Mortality percentages of tobacco budworm larvae on leaves collected from designated rows in swath width								
	16-row			20-row			24-row		
	1–6[1]	7–8	11–16	1–6[1]	9–10	14–20	1–6[1]	11–12	17–24
0	100	100	100	100	100	100	100	100	100
1	100	100	100	96	100	100	96	100	100
3	64	60	65	69	64	60	51	84	56
5	28	43	33	53	18	29	30	30	25
6	32	41	40	31	40	40	27	28	41
8	13	20	0	11	9	13	16	0	4

[1] Rows 1 to 6 in SE portion of plot.

most samples, and care was taken to maintain a "primed" column.

Quantitative determinations were made by comparing standard curves of concentration (in grams) to peak height. The standard curve for methyl parathion showed a linear range of 3×10^{-9} to 0.4×10^{-9} and a lower limit of sensitivity of 0.4×10^{-9}.

Concentrations were expressed as parts per million and percentage on the leaf surface as micrograms per square centimeter and were not corrected for percentage of recovery. Number of days for 50-percent loss was determined by calculating percentage loss after 4 days.

To determine recovery rates of methyl parathion, homogenized (with 100 ml. redistilled or nanograde benzene) cotton leaves (25 or 50 grams) were spiked with various concentrations of methyl parathion. Homogenates were then filtered through sodium sulfate on filter paper (Whatman No. 1) into a bottle containing 1 to 4 grams Nuchar C-190 or Nuchar-Attaclay (from Varian-Aerograph) (1:1 ratio) for 10 to 20 minutes. The benzene was then filtered into another vial through 2 to 3 grams of sodium sulfate and evaporated to dryness with air. The dry extract was then redissolved in 1 to 5 ml. benzene for analysis. Recoveries were 75 to 92 percent (table 4). About 4 hours were required to determine 10 recoveries simultaneously.

The conditions and materials for the gas chromatograph systems for determination of methyl parathion were as follows:

(1) Research Specialties Model 600 (now Warner Chillicott Instruments Division) having electron-capture detection with a 10-millicure strontium-90 source; ¼-inch × 4-foot per glass column or ¼-inch × 2-foot aluminum column packed with 10 percent DC 200 on Gas Chrom Q 80/90 mesh support; 105 ml. per minute total flow of scavenger plus carrier, of which 30 ml. per minute was scavenger flow of prepurified nitrogen; temperatures of the injection port, column, and detector, respectively, held isothermal at 230°, 210°, and 250° C.; electrometer sensitivity of 4.1×10^{-11} amp. for full-scale pen deflection; and a chart speed of 0.5 inch per minute; or

(2) Varian-Aerograph gas chromatograph Model 600-D, having a phosphorus detector

TABLE 4.—*Percentages of malathion and methyl parathion recovered from cotton foliage by gas chromatographic techniques*

Insecticide	Concentration	Recovery (± standard deviation)
	P.p.m.	*Percent*
Malathion[1]	3.0	98.9
	50.0	94.0
	100.0	93.3
	200.0	90.0
	232.0	[2]94 ± 4.9
Methyl parathion	3.0	[3]75 ± 0.99
	3.0	[4]92 ± 1.5

[1] Average of two analyses; determined on Micro-Tec Model 2500.

[2] Average and standard deviation of 22 analyses; determined on Research Specialties Model 600.

[3] Average of 10 analyses; determined on Research Specialties Model 600.

[4] Average of 10 analyses; determined on Varian-Aerograph Model 600-D with phosphorus detector.

(consisting of a flame detector with salt tips which were changed and cleaned for reuse as sensitivity decreased); 1/8-inch × 3- or 5-foot glass column packed with 10 percent DC 200 on Gas Chrom Q 80/90 mesh support, 18 ml. per minute flow of hydrogen, 170 ml. per minute of breathing air, and 18 ml. per minute of prepurified nitrogen; temperatures of injection port, column, and detector were 250°, 200°, and 200° C.: electrometer settings were 1× and 2× (1×10^{-12} amp.) for full-scale pen deflection; and a chart speed of 0.5 inch per minute.

Samples consisted of disks cut out of leaves of cotton plants with a hand punch. Each disk was 3.19 cm. in diameter or 8 cm.^{2} and ranged in weight from 0.615 to 0.036 gram. The disks were taken from leaves fully exposed to the sprays in the laboratory or field on indicated days after application and brought to the laboratory for extraction of residues. No attempt was made to select leaves randomly from treated plants. Chloroform or water (10 or 50 ml.) was added to the disks (5 or 50) in a 100-ml. screw-cap bottle.

The bottle containing the chloroform and leaf disks was shaken for 15 minutes on a mechanical shaker and decanted through a plug of glass wool into a graduated cylinder. The chloroform was then poured into a 250-ml. beaker and evaporated to dryness. The residue was then dissolved in benzene or acetone for analysis.

Water extraction was made by placing the leaf disks in the screw-capped bottle with water and shaking it for 15 minutes on a mechanical shaker. It was then filtered through a plug of glass wool into a separatory funnel with about 3 grams sodium chloride to prevent formation of an emulsion. The extract was then washed twice with an equal volume of benzene. The benzene was then filtered (Whatman No. 1) into another screw-capped bottle and evaporated to dryness at room temperature. Following evaporation, the dry extract was redissolved in 1 to 5 ml. benzene or acetone for analysis.

The disks from the chloroform or water washes were then homogenized in a blender with 50 ml. benzene and 1.5 to 2.0 grams decolorizing carbon (Nuchar) or Nuchar-Attaclay; then the homogenate was filtered. The benzene was evaporated and the dry extract redissolved in 1 ml. or more benzene or acetone as described above for analysis.

Total residues of methyl parathion immediately after it was applied at 1.0 pound per acre were greater for ULV than for LV sprays (table 5). However, residues of methyl parathion were greater on the leaf surface when it was applied in LV than in ULV sprays.

In field tests the number of leaf disk samples taken on indicated days, type of application, equipment used, nozzle type, and total solution applied are summarized in table 6 and its footnotes. Airplane applications were made 3 to 6 feet above the ground.

TABLE 5.—*Residues of methyl parathion from leaf surface and leaf homogenates applied in ULV and LV sprays in the laboratory*

Days after application	Methyl parathion residues from chloroform extract		
	From leaf surface and homogenates	From leaf surface only	
	P.p.m.	*Percent*	*µg/cm²*
ULV (*1.0 lb./acre*):			
0	84.4	57	3.60
2	4.2	50	0.15
4	5.3	60	.23
ULV (*0.5 lb./acre*):			
0	5.5	58	.45
2	4.2	50	.30
4	4.2	50	.30
LV (*1.0 lb./acre*):			
0	51.1	88	3.37
2	8.6	60	0.38
4	7.8	72	.40
LV (*0.5 lb./acre*):			
0	27.2	85	1.73
2	3.9	67	0.15
4	5.8	57	.23

At Altamira, Mexico, more methyl parathion was found initially and 6 days after it was applied in ULV than in LV sprays. The chloroform wash recovered 22 to 57 percent more methyl parathion following ULV and LV sprays than the water wash on the day of application.

At Bayview, approximately 87 percent of methyl parathion was found in or on the cotton leaf 7 days after being applied in a ULV spray; however, 85 percent of this total was recovered from the leaf surface in the water wash. Ten and 13 days after application, methyl parathion was recovered with water wash from the leaf surface but none was recovered with the chloroform wash.

Three experiments were conducted at Brownsville. In 1966 (experiment 1), about 50 percent of the ULV spray (1.25 pound per acre) was lost 2 and 5 days after the first and second applications, respectively, with the chloroform wash; about 3 and 15 percent were recovered with the water wash. Chloroform washes removed all methyl parathion from the leaf surface; 17 percent or less was removed with water washes. In the second experiment in 1966, methyl parathion deposits were reduced 58 to 82 percent after 5 days following two consecu-

TABLE 6.—*Residues of methyl parathion from cotton leaf surfaces and leaf homogenates following ULV and LV sprays in the field*

Days after application	Methyl parathion residues					
	Water wash		Chloroform wash		Amount per cm.[2] on leaf surface	
	Total[1]	On leaf surface	Total[1]	On leaf surface	Water wash	Chloroform wash
	P.p.m.	*Percent*	*P.p.m.*	*Percent*	*µg.*	*µg.*
ALTAMIRA, MEXICO, 1965						
ULV *(1.0 lb./ acre)*[2]:						
First application:						
0	71.8	8	67.7	56	5.0×10^{-4}	3.1×10^{-3}
1	25.9	10	26.2	80	2.0×10^{-4}	1.7×10^{-3}
6	6.4	9	21.2	95	5.0×10^{-5}	1.6×10^{-3}
Second application:						
0	64.7	5	75.6	71	2.8×10^{-4}	4.3×10^{-3}
1	26.4	0	64.1	59	0	3.0×10^{-3}
6	8.3	0	20.5	75	0	1.2×10^{-3}
ULV *(1.5 lb./ acre)*[2]:						
First application:						
0	95.0	11	56.1	95	8.5×10^{-4}	4.3×10^{-3}
1	46.9	6	39.0	87	2.4×10^{-4}	2.7×10^{-3}
6	12.6	11	19.5	91	1.1×10^{-4}	1.4×10^{-3}
Second application:						
0	38.4	20	60.5	91	6.3×10^{-4}	4.5×10^{-3}
1	23.7	5	33.8	100	8.9×10^{-5}	2.7×10^{-3}
6	0	0	0	0	0	0
LV *(1.0 lb. / acre)*[2]:						
First application:						
0	38.7	36	44.8	94	1.1×10^{-3}	3.4×10^{-3}
1	11.9	21	22.3	96	2.0×10^{-4}	1.7×10^{-3}
6	0	0	10.0	95	0	7.7×10^{-4}
Second application:						
0	24.2	0	33.7	95	0	2.6×10^{-3}
1	7.8	30	21.1	88	1.3×10^{-4}	1.5×10^{-3}
6	5.4	0	24.6	87	0	1.7×10^{-3}
BAYVIEW, TEXAS, 1966						
ULV *(1.0 lb. / acre)*[3]:						
0	165.8	3	67.4	87	7.5×10^{-2}	3.6×10^{-1}
7	52.3	83	14.0	86	1.3×10^{-1}	3.7×10^{-2}
10	20.3	51	25.0	0	4.5×10^{-2}	0
13	10.0	20	25.0	0	2.3×10^{-2}	0
BROWNSVILLE, TEXAS EXPERIMENT 1—1966						
ULV *(1.25 lb. / acre)*[4]:						
First application:						
0	365.1	1	595.4	100	1.7×10^{-4}	4.8×10^{-2}
2	9.0	0	220.1	100	0	1.8×10^{-2}
7	0	---------	17.1	100	0	1.4×10^{-2}
Second application:						
0	151.4	1	527.1	100	1.1×10^{-4}	4.3×10^{-2}
5	22.0	0	298.1	100	0	2.4×10^{-2}
9	0	---------	193.1	100	0	1.6×10^{-2}

See footnotes at end of table.

TABLE 6.—*Residues of methyl parathion from cotton leaf surfaces and leaf homogenates following ULV and LV sprays in the field*—Continued

Days after application	Methyl parathion residues					
	Water wash		Chloroform wash		Amount per cm.[2] on leaf surface	
	Total[1]	On leaf surface	Total[1]	On leaf surface	Water wash	Chloroform wash
	P.p.m.	*Percent*	*P.p.m.*	*Percent*	*µg.*	*µg.*
EXPERIMENT 2—1966						
LV *(1.0 lb. / acre)*[5]:						
First application:						
0	(6)	(6)	31.0	97	(6)	10.50
1	(6)	(6)	22.0	89	(6)	7.50
5	(6)	(6)	14.0	100	(6)	5.25
Second application:						
0	(6)	(6)	56.0	69	(6)	13.12
2	(6)	(6)	19.0	100	(6)	7.13
5	(6)	(6)	9.0	100	(6)	3.38
EXPERIMENT 3—1967						
ULV *(1.0 lb. / acre)*[7]:						
First application:						
0	(6)	(6)	2.14	7	(6)	5.0×10^{-2}
1	(6)	(6)	2.00	25	(6)	3.8×10^{-2}
2	(6)	(6)	1.23	82	(6)	5.8×10^{-3}
3	(6)	(6)	0.14	100	(6)	3.5×10^{-3}
6	(6)	(6)	0	—	(6)	0
8	(6)	(6)	0	—	(6)	0
Second application:						
0	(6)	(6)	1.0	100	(6)	0
1	(6)	(6)	0	0	(6)	0
6	(6)	(6)	0	0	(6)	0
LV *(1.0 lb. / acre)*[5]:						
First application:						
0	(6)	(6)	0.75	80	(6)	1.5×10^{-2}
1	(6)	(6)	.44	45	(6)	6.0×10^{-3}
2	(6)	(6)	.55	64	(6)	5.0×10^{-3}
3	(6)	(6)	.37	68	(6)	3.0×10^{-3}
6	(6)	(6)	.21	71	(6)	1.5×10^{-3}
8	(6)	(6)	.21	71	(6)	1.5×10^{-3}
Second application:						
0	(6)	(6)	.62	84	(6)	0
1	(6)	(6)	.06	100	(6)	0
6	(6)	(6)	.04	100	(6)	0

[1] Residue from both leaf surface and leaf homogenates.

[2] ULV sprays applied with Piper Pawnee airplane equipped with Mini-Spin nozzles; LV sprays applied with Piper Pawnee airplane equipped with fan nozzles at 3 gallons per acre; 50 leaf disks per sample and two samples per analysis; plots were 1 to 5 acres in size.

[3] Applied with Snow airplane (Model A or C) equipped with Mini-Spin nozzles at 1 quart per acre; 50 leaf disks per sample and two samples per analysis; plots 1 to 10 acres in size.

[4] Applied with Snow airplane (Model A or C) equipped with Mini-Spin nozzles at 1.25 quarts per acre; 50 leaf disks per sample and two samples per analysis; plots 1 to 5 acres in size.

[5] Applied with a Hahn Highboy equipped with 3× nozzles spaced 20 inches apart at 5 gallons per acre; 10 leaf disks per plot (1/10 acre) for 4 replicates for a total of 40 leaf disks per analysis.

[6] Water wash of cotton leaves not made.

[7] Applied with a Hahn Highboy equipped with a John Blue ULV sprayer; plots (1/10 acre) for four replicates for a total of 40 leaf disks per analysis.

tive applications of LV sprays; after 1 and 2 days, 35 and 62 percent, respectively, were lost.

In 1967 (experiment 3), ULV sprays of methyl parathion showed 2 to 3 times more residue than LV sprays after two applications using a Highboy sprayer. However, more was found on the leaf surface after LV than after ULV sprays.

In experiments where both ULV and LV applications of methyl parathion were made, deposits immediately following these sprays were compared; 58.6 percent (35.5 to 65.0 range) more toxicant was found after ULV than after LV sprays. Fifty percent of methyl parathion was lost after 2.8 days when applied in ULV sprays; 50 percent was lost after 2 days when applied in LV sprays.

A comparison was made of methyl parathion applied (micrograms per square centimeter) on the upper leaf surface with that theoretically applied at 1 pound per acre in laboratory and field experiments. First, the deposits in the chloroform wash (table 6) were recorded within 2 hours after application. These deposits were then related to concentrations theoretically applied to the leaf.

The theoretical deposit of methyl parathion at 1 pound per acre was 11.2 μg/cm.^{2}. An average of 17 percent of the theoretical concentrations of methyl parathion was recovered from ULV and LV sprays. The percentages of recovery (of that theoretically applied) ranged from 117 to 0.03 from 6 of the 11 sets of data (where both LV and ULV sprays were applied in the same experiment). These data were summarized from all experiments.

Deposits of malathion in field tests

Residues of malathion were determined by the GLC methods described for methyl parathion. Range of linearity was 2.0×10^{-9} to 4.5×10^{-9} grams, and lower limits of sensitivity were 2.0×10^{-9} grams. The R_t of aldrin to malathion was 3 to 6 minutes under the operating conditions described for both gas chromatographs. Malathion concentrations are expressed as described for methyl parathion. Recoveries of malathion were 94 to 98 percent (table 4); they were determined by the methods described for methyl parathion.

The gas chromatograph system No. 3 used to determine malathion residues in the Progreso experiment was a Micro-Tek gas chromatograph Model GC-2500 having an electron-capture detector with a 220-millicurie tritium source and parallel-plate detector; ¼-inch × 6-foot aluminum column packed with 10 percent DC 200 oil on Anakrom ABS 80/90 mesh support; 120 ml.-per-minute flow rate of prepurified nitrogen, scavenger plus carrier; temperatures of the injection port, column, and detector, respectively, held isothermal at 230°, 210°, and 190° C.; electrometer sensitivity of 10^{-9} amp. for full-scale pen deflection; and a chart speed of 0.3 inches per minute. A removable Pyrex insert was placed in the inlet with essentially "on column" injection. Gas chromatograph system No. 1 as described for determining methyl parathion residues was used for the Brownsville experiment.

Extraction of residues from leaf surfaces was made with chloroform or water at Progreso. At Brownsville only the chloroform extract was made. Chloroform extraction was made as described for methyl parathion. Water extraction was made by placing a separate leaf disk sample from that extracted with chloroform in a beaker with 100 ml. of water. The beaker was then shaken gently for 15 minutes on a mechanical shaker. The extract was then filtered through a plug of glass wool into a 250-ml. separatory funnel with approximately 3 grams sodium chloride (to prevent formation of an emulsion) and washed with four 20-ml. portions of benzene. One-fourth of the recovered benzene was evaporated slowly (at not over 70° C.) to dryness on a steam bath with a current of dry air. The dry extract was then redissolved in 1 to 5 ml. of benzene for analysis.

The leaf homogenates were made in the same manner as the chloroform extraction for methyl parathion. Malathion residues in the leaf homogenates after the water wash were determined by difference between the sum of the residue from the chloroform wash and its leaf homogenate. We assumed that this sum was the maximum we could obtain from the leaf.

At both rates, more malathion was applied as a LV spray than as a ULV spray, based on initial concentration (table 7). This is contrary to the findings of Wheeler et al. (*17*) and Nemec et al. (*14*) with malathion, methyl parathion, and Azodrin (3-hydroxy-*N*-methyl-*cis*-crotonamide dimethyl phosphate). The probable explanation for our finding is the difference in altitude at which the sprays were applied. The ULV applications were made from a height of 10 feet and the LV sprays from 6 feet.

Leaves sprayed with malathion were bioassayed (table 8) as described earlier for the Progreso experiment. The residues were expressed in micrograms per square centimeter. The two middle rates had larval mortalities between those for the low or high rates.

These data indicate that 1.51 μg./cm.^{2} or more malathion is required to kill 98 percent or more bollworms or tobacco budworms.

The number of days required for 50-percent loss of malathion is shown in table 9. The residue data from the Progreso experiment (table

TABLE 7.—*Malathion recovered from cotton leaf surface and in leaf homogenates*

Days after application	Recovery of malathion: From leaf surface and homogenates	From leaf surface only: In water wash	From leaf surface only: In chloroform wash	Amount/cm.²: In water wash	Amount/cm.²: In chloroform wash
	P.p.m.	*Percent*	*Percent*	*µg.*	*µg.*
PROGRESO, TEX. 1965					
ULV *(1.25 lb./acre)*:					
First application:					
0	266	88	90	3.02	3.06
2	49	76	81	0.49	0.52
4	16	55	80	.10	.17
Second application:					
0	249	75	92	2.41	2.93
2	119	78	91	1.22	1.40
4	104	82	84	1.11	1.10
Fourth application:					
0	174	86	85	1.86	1.86
2	17	7	52	0.01	0.11
4	12	36	56	.05	.09
LV *(1.25 lb./acre)*:					
First application:					
0	246	84	88	2.10	2.69
2	31	59	66	0.24	0.26
4	20	19	44	.04	.11
Second application:					
0	1,073	80	92	12.10	12.56
2	496	72	85	4.79	0.54
4	80	33	78	0.36	.79
Fourth application:					
0	961	84	87	10.30	10.23
2	108	67	71	0.94	1.01
4	22	52	73	.15	0.21
ULV *(2.5 lb./acre)*:					
First application:					
0	307	88	90	3.66	3.50
2	49	59	83	0.37	0.51
4	21	34	71	.06	.19
Second application:					
0	531	86	95	5.99	6.44
2	236	78	91	2.40	2.79
4	206	54	87	1.54	2.37
Fourth application:					
0	259	89	85	2.80	2.71
2	18	2	62	0.01	0.17
4	14	5	59	.01	.12

TABLE 7.—*Malathion recovered from cotton leaf surface and in leaf homogenates*—Continued

Days after application	Recovery of malathion: From leaf surface and homogenates	From leaf surface only: In water wash	From leaf surface only: In chloroform wash	Amount/cm.²: In water wash	Amount/cm.²: In chloroform wash
	P.p.m.	*Percent*	*Percent*	*µg.*	*µg.*
LV *(2.5 lb./acre)*:					
First application:					
0	435	89	92	3.60	5.06
2	79	77	77	0.77	0.76
4	23	67	59	.19	.18
Second application:					
0	736	80	91	2.69	8.50
2	483	71	86	4.31	5.18
4	99	43	67	0.57	0.85
Fourth application:					
0	624	85	86	6.64	6.64
2	90	40	57	0.47	0.50
4	35	46	65	.23	.31
BROWNSVILLE, TEX. 1966					
ULV *(2.5 lb./acre)*:					
First application:					
0	262	(1)	8	(1)	6.49
1	97	(1)	3	(1)	2.35
2	89	(1)	4	(1)	2.13
3	32	(1)	11	(1)	0.70
6	5	(1)	38	(1)	.08
8	0.6	(1)	100	(1)	.02
Second application:					
0	110	(1)	1	(1)	2.74
1	5	(1)	12	(1)	0.11
6	3	(1)	46	(1)	.04
LV *(2.5 lb./acre)*:					
First application:					
0	117	(1)	3	(1)	2.83
1	91	(1)	4	(1)	2.19
2	45	(1)	12	(1)	0.99
3	16	(1)	17	(1)	.34
6	13	(1)	14	(1)	.28
8	3	(1)	39	(1)	.04
Second application:					
0	72	(1)	3	(1)	1.73
1	9	(1)	19	(1)	0.19
6	1	(1)	75	(1)	.01

[1] Water wash of cotton leaves not made.

TABLE 8.—*Mortality of tobacco budworm and bollworm larvae in relation to amount of malathion residue on leaf surface*

Range in residue (µg./cm.²)	Mortality	
	Mean	Range
	Percent	*Percent*
0.10–0.50	30	0– 94
0.51–0.99	69	54– 85
1.00–1.50	52	32– 79
1.51	98	65–100

TABLE 9.—*Days required for 50 percent loss of malathion from the leaf surface*

Malathion (lb./acre)	No. of days for 50 percent loss after application		
	1	2	4
ULV spray:			
1.25	0.81	3.45	2.15
2.5	2.15	3.28	2.11
LV spray:			
1.25	2.17	2.15	2.04
2.5	2.11	2.30	2.12

7) were used to determine the days. Malathion residues from ULV sprays persisted longer after four of the six applications than the LV sprays.

In Brownsville more malathion was found initially when it was applied in ULV than in LV sprays (table 7). More residue was found 6 and 8 days after the first application of LV spray, but not the second. Generally, the amount of malathion found on the leaf surface decreased with time. Amounts of malathion in LV sprays applied by ground equipment in the Brownsville experiment were much lower than in sprays applied by airplane in the Progreso experiment. These data suggest that more malathion is applied by airplane than by ground equipment at equal rates.

Residues of malathion were compared with those resulting from theoretical applications in the Brownsville and Progreso experiments. Residues of malathion (µg./cm.²) were determined on both sides of the leaf surface in the chloroform wash within 2 hours after application. Theoretically, 28 and 56 µg./cm.² of malathion on both leaf sides should be deposited when 1.25 and 2.5 pounds per acre are applied, respectively. An average of 14 percent (5 to 44 percent) of the malathion theoretically applied was found immediately after application in the Progreso experiment, and 8.4 percent was found after ULV (11.6 percent) and LV (5.1 percent) sprays in the Brownsville experiment.

Drift of methyl parathion in field tests

Oil-sensitive cards and filter-paper samples were collected after airplane applications of methyl parathion at 1 pound per acre in ULV and LV sprays at heights of 5 and 20 feet. A Snow airplane was used. The ULV spray boom had 8 Mini-Spin nozzles, as described for the swath-width experiment, and was calibrated to deliver 1 quart per acre. The LV spray boom had fan nozzles calibrated to deliver 3 gallons per acre. The sprays were applied in a crosswind on a southeast to northwest airport runway. Spray particles (number and size of particle) were determined from oil-sensitive cards (Home & Farm Chemicals, Charlotte, N. C.). Amounts of methyl parathion were determined from filter papers by gas chromatographic methods with GLC system No. 1.

Before each spray application, oil-sensitive Sudan Red 4 x 5-in. cards were taped on a runway 75 feet wide. At each of two sites, one filter paper (Whatman No. 1) was placed in a 9-cm. petri dish 12 to 6 inches away from a card. Both the cards and the filter paper were placed beneath the plane, upwind and downwind. Each site was 15 feet from the edge of the runway and the sites were 45 feet apart. The plane made three passes over the same location. After each application the cards and the petri dishes were collected and placed in boxes for transporting to the laboratory.

To relate the droplet size to spot size on the cards, it was first necessary to determine the "spread factor" of methyl parathion from droplets of known diameter. Glass rods (0.5-1.0 mm. inside diameter) drawn with heat to various diameters were prepared and dipped into the emulsifiable concentrate formulation. After the rods were withdrawn, the excess liquid was allowed to drip off. Under 10× microscopic observation, the rod was tilted towards the vertical to permit the retained liquid to flow to the tip, forming a droplet. Before the droplet was placed on the paper, the area immediately above the tip was wiped gently with absorbent paper to eliminate any retained liquid. Before the droplet was placed on the card it was measured with a calibrated eyepiece on a binocular microscope. The droplet was then placed on a card and its diameter again measured. The spots on the cards were then remeasured after 24 hours to determine the spread factor. For each droplet, the difference (in microns) between the 0-

and 24-hour measurements was the spread factor. Sixty-three droplets of 200- to 1,450-micron size were used to determine this spread factor. The wind velocity averaged 14.2 m.p.h. from the SE during the test period.

The droplet size in microns, percentage of card covered with droplets, and nanograms of methyl parathion per cm.^{2} on filter paper at given distances from the center of the swath are presented in table 10. Amounts were ad-

TABLE 10.—*Recovery of methyl parathion applied by airplane in ULV and LV sprays at various distances from center of swath at heights of 5 and 20 feet, Bayview, Tex.*

Distance from center of swath	LV spray			ULV spray		
	Droplet size	Card covered	Methyl parathion per cm.^{2} of filter paper	Droplet size	Card covered	Methyl parathion per cm.^{2} of filter paper
Feet	μ.	*Percent*	*Ng.*	μ.	*Percent*	*Ng.*
			HEIGHT 5 FEET			
830	0	0	0.68	127.1	24×10^{-6}	0
630	0	0	2.16	144.3	47×10^{-6}	1.78
430	0	0	0.28	88.8	70×10^{-6}	3.35
230	0	0	1.02	78.1	54×10^{-6}	0.29
180	0	0	0.84	87.9	21×10^{-5}	1.04
130	55.7	14×10^{-5}	0	77.5	38×10^{-5}	1.07
80	68.0	11×10^{-6}	.10	79.8	26×10^{-4}	1.04
30	71.0	33×10^{-6}	.46	76.5	68×10^{-4}	1.68
20	71.1	98×10^{-6}	.45	84.4	71×10^{-4}	6.08
10	100.2	17×10^{-5}	0	88.1	11×10^{-3}	3.30
0	97.9	51×10^{-4}	0	93.9	78×10^{-4}	15.00
10	66.1	37×10^{-6}	1.46	74.5	56×10^{-4}	1.00
20	72.5	14×10^{-5}	0	71.7	20×10^{-4}	0.85
30	51.1	85×10^{-6}	0.33	66.7	26×10^{-4}	.40
80	0	0	0	25.5	48×10^{-6}	.22
130	36.2	10×10^{-6}	.34	0	0	2.94
180	0	0	0	0	0	1.36
230	0	0	0	0	0	0
430	0	0	0	0	0	0.81
630	0	0	0.33	0	0	1.39
830	0	0	0	0	0	0.46
			HEIGHT 20 FEET			
860	----------	----------	0	27.9	19×10^{-6}	1.10
660	127.5	95×10^{-6}	0	46.5	85×10^{-7}	0.76
460	----------	----------	0	81.8	11×10^{-5}	.98
260	106.7	40×10^{-6}	0	56.3	33×10^{-4}	1.34
210	77.8	79×10^{-6}	1.52	54.3	48×10^{-3}	0.18
160	----------	----------	0	69.8	57×10^{-4}	.34
110	73.7	90×10^{-5}	0.12	69.1	11×10^{-3}	4.11
60	80.4	12×10^{-3}	.31	70.8	68×10^{-4}	0.37
40	91.6	51×10^{-6}	0	67.3	91×10^{-4}	.50
20	----------	----------	0	71.5	70×10^{-4}	.14
0	139.6	12×10^{-5}	1.34	71.2	80×10^{-4}	.11
20	106.4	90×10^{-6}	0	78.7	63×10^{-4}	.31
40	0	0	0.97	0	0	0
60	0	0	.60	0	0	0
110	0	0	0	0	0	0
160	0	0	2.91	0	0	0
210	0	0	0	0	0	0
260	0	0	0.13	0	0	0.18
460	0	0	.22	0	0	0
660	0	0	0	0	0	0
860	0	0	0.33	0	0	0

justed to a single swath because only one pass would be made to a location during a normal spray operation. The droplet size and percentage of card covered were adjusted for the spread factor from the initial droplet size.

More methyl parathion drifted from heights of 5 and 20 feet in ULV than in LV sprays. No droplets from LV sprays at the 5-foot height were found at a distance greater than 130 feet from the center of the treated area, whereas droplets (corrected for a spread factor of 7.02) were found 800 feet from the center of the target area from ULV sprays. The most important finding was that 5.4 times more methyl parathion (at all locations) was found after ULV than after LV sprays at either height. More methyl parathion also was found directly beneath the plane after ULV sprays at both heights.

CONCLUSIONS

Methyl parathion and malathion were more effective when applied in ULV than in LV sprays against larvae of the bollworm and tobacco budworm. Methyl parathion applied in both spray methods was effective against pink bollworm in field experiments, but malathion was not.

GLC techniques proved to be useful in determining deposits of malathion and methyl parathion on cotton foliage.

Generally, greater deposits were found following ULV sprays of methyl parathion than LV sprays. More methyl parathion drifted from heights of 5 and 20 feet in ULV than in LV sprays.

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